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CHEMICAL LAND HOLDINGS, INC.

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U.S. Environmental Protection Agency, Region II Emergency and Remedial Response Division 290 Broadway, 19th Floor, Room W-20 New York, NY 10007-1866

Attention:

Ms. Janet Conetta

Strategic Integration Manager

Subject:

Results of Centrifuge Comparison Test

Passaic River Study Area

Administrative Order on Consent Index No. II-CERCLA-0117

Dear Ms. Conetta:

Please find attached a memorandum from BBL reporting on the results of a comparison test between a continuous-flow centrifuge and a multiplate filtration system. This comparison was conducted in relation to the Combined Sewer Overflow (CSO) program that is pending under the Ecological Sampling Plan (ESP) of the Passaic River Remedial Investigation/Feasibility Study (RI/FS).

During the CSO Trial Run two major issues were noted when filtering the water to obtain solids:

- High water content the water contained within the filters was having an adverse impact on the analytical results, resulting in qualified data
- Time filtration of the large volume of water required to obtain sufficient solids mass was requiring excessive time and labor

As a result of these issues, CLH considered a number of mitigating actions, and proceeded with a comparison between the centrifuge and traditional filtration. The purpose of this letter is to provide the attached and, based on the superior performance of the centrifuge, recommend that the field centrifuge be used to collect solids from the CSO discharge instead of the multiplate filter. Since this represents a departure from the ESP, CLH is submitting this as a formal recommendation, and requests your prompt concurrence.

Background

Standard Operating Procedure (SOP) 6 of the ESP specifies that, if necessary, the CSO discharge water will be filtered through a $1.2\mu m$ filter to collect solids. Due to the large volume of water to be processed, a multi-plate filter was used to enable several filters to be coated with solids at the same time. During the CSO Trial Run task, it was taking several person-days to process the

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required water volume. In addition, the water content of the samples was high, requiring the laboratories to qualify the analytical results due to high moisture content.

To rectify these issues, CLH considered a number of mitigation steps:

- Pump air or inert gas across the filters at the conclusion of each filter set. Based on
 experience, this would not reduce moisture content sufficiently, and would require even more
 labor/time to conduct the activity.
- Filter-press. This approach was judged to be promising to reduce moisture content, but would potentially cause the loss of solids and was particularly labor intensive.
- Continuous-flow centrifuge. This was judged to have the greatest potential to both reduce moisture content and labor/time for processing.

Results

CLH evaluated the performance of a portable contribuge versus filtration to remove solids from a large volume of water. The two systems were compared for performance (i.e., the distribution of grain sizes removed) and for labor/time for processing. To conduct the evaluation, 400 gallons of Passaic River water were collected and kept homogenized with a power stirrer while collecting samples via the two methods. The attached memorandum from BBL provides additional detail on the process, the tests conducted, and the results.

Filtration was conducted with a single multiplate filter system (6 filters) and centrifugation was performed with a Westfalia centrifuge as modified by Machine All, Inc. of Ontario, Canada. Particle Technology Laboratories (Downers Grove, IL) analyzed the sediment samples for grain size distribution.

The attached table summarizes the performance characteristics of the two systems. The pertinent results are:

- Grain size distribution is essentially the same.
- Water content of the centrifuged sample is ~2/3 of the filtered sample a significant improvement.
- The time/labor required for the centrifuge is less than half of that required for filtration.

Based on these results, CLH is recommending that the centrifuge be used in lieu of filtration. The attached SOP 6 has been modified to allow the option for centrifugation, and to detail appropriate procedures to implement this method.

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Please indicate the Agency's concurrence with this improvement in writing.

If you have any questions, please contact me at (757) 258-7720.

C. Leistenler

Clifford Firstenberg

Project Manager

On behalf of Occidental Chemical Corporation

(as successor to Diamond Shamrock Chemicals Company)

attachment

- 1. BBL Memorandum
- 2. Revised SOP 6





To:

Cliff Firstenberg

Chemical Land Holdings, Inc.

From:

Russell Houck

Re:

Centrifuge Demonstration

09994.104

Date:

05/30/00

cc:

Tony Wolfskill Robert Romagnoli

Lowell McBurney

Joe Molina

Doug Rusczyzk

Sarah Hill

This memorandum generally describes the centrifuge demonstration program conducted at the BBL Kearny field office between May 16 and May 19, 2000. At the request of CLH, this program was developed to evaluate the performance of the Westfalia centrifuge produced by Machine-All, Inc, Mississauga, Ontario, Canada.

Water Collection

On May 16, 2000, BBL collected 400 gallons of sediment-laden water from the Passaic River for use in the centrifuge demonstration. To collect the water, BBL transported equipment from the Kearny field office to the Kearny boat launch. At the hoat launch area, two submersible pumps were placed within the Passaic River in approximately 1 to 2 feet of water. Pumps were placed inside perforated 5-gallon buckets to prevent intake of debris. During pumping, BBL personnel agitated river sediments in the area of the pumps to promote intake of suspended sediments. Once collected, water was then transported back to the Kearny field office for temporary storage.

Filterable Solids Collection

On May 18, 2000, BBL utilized a portion of the collected water to obtain a filterable solids sample for particle size analysis. The results from this test would help in evaluating the solids capture efficiency of the centrifuge. BBL used a 3-plate (6 filter) AXYS multiplate filtration system and peristaltic pump to collect the sample. Prior to filtering, the AXYS multiplate was deconned and the 400 gallon tank of river water was mixed using a power drill and long-stern paddle mixing tool. Water was kept mixed during the process by intermittently using the power mixer and a boat oar. Over the course of 49 minutes, approximately 13 1/2 gallons of water were filtered, and three sets of six filters (18 total) were collected. All filters (containing a heavy black/green/brown film of wet solids) were placed in two 1-liter glass jars, labeled (sample ID: FILTSED01), and stored for shipment on the following day. Filtrate was collected and placed into 55-gallon storage drums and returned to the Passaic River the following day.

Centrifuge Demonstration

On May 19, 2000, BBL and Mr. Joe Kania of Machine-All, Inc. conducted a demonstration of the Westfalia centrifuge. The purpose of the demonstration was to test the system's ability to separate solids from water (especially fines). To conduct the demonstration, BBL/Machine-All set up the centrifuge system in the Kearmy field office garage area. Initially, Mr. Kania instructed BBL personnel in the set-up, use and disassembly of the Westfalia centrifuge unit. Once CLH personnel (C. Firstenberg, T. Wolfskill) arrived on site, Mr. Kania, with BBI, assistance, conducted a demonstration using approximately 20 grams of clay mixed with approximately 35 gallons of tap water. For this initial demonstration, the centrifuge operated at a rate of approximately 2 to 5 L/minute (average approximately 3.5 L/min) for approximately 35 minutes. Flow rate was checked and adjusted several times during the demonstration.

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Following shutdown of the centrifuge and pump, the bowl assembly was removed and disassembled by Mr. Kania. A thin brown clay layer was visible on the inner bowl surfaces. Water and clay particles also remained in the base bowl. Several grams of wet sediment were scraped from the surface of one of the bowls. The bowl assembly was deconned and replaced for a second demonstration.

The centrifuge system was then set up to draw from the 400-gallon tank of river water. Prior to operating the centrifuge, the 400-gallon tank was mixed using the power drill/mixer and boat oar. During this second demonstration, the centrifuge ran for 50 minutes at a flow rate of approximately 1 to 2 L/minute (the flow rate was checked periodically at the discharge). Approximately 15 to 20 gallons of water was discharged from the centrifuge during the second demonstration. The bowl assembly was removed and disassembled.

A sediment layer of noticeable thickness was visible on the inner bowl surfaces. Sediment was black/green/brown in color. Water and sediment particles also remained in the base bowl. Using a plastic spatula, wet sediment was scraped from the surface of the bowls. Collected sediment was placed in a 250 ml glass jar (sample ID CENTSED01) and weighed. Total net weight of the collected sediment (wet) was 64.1 grams. The bowl assembly was then deconned.

Water discharged from the centrifuge was collected and returned to the 400 gallon tank. Water from collection of the filterable solids sample was also returned to the 400- gallon tank. The tank was then transported to the Kearny boat launch where the water was pumped back into the Passaic River.

The filterable solids sample (collected May 18, 2000) and the centrifuge sediment sample (collected May 19, 2000 during the second demonstration) were shipped via Fedex to Particle Technology Laboratories (PTL), Downers Grove, IL, for particle size analyses. In addition, the centrifuge sample was to be analyzed for moisture content.

Laboratory Results

On May 23, 2000, PTL faxed to BBL results from the particle size testing and moisture content analysis. Both the filterable solids sample (FILTSED01) and the centrifuge sample (CENTSED01) were analyzed for particle size using a Malvern S Laser particle size analyzer which utilizes a laser diffraction sizing technique. Histogram and tabular data from the particle size analyses are attached. Results show that, in general, the two sampling methodologies (filtering and centrifuge) captured similar fractions of particle sizes. A portion of the results are summarized below.

Particle size (um)	Filterable Solids Volume under (%)	Centrifuged Solids Volume under (%)	
0.522	3.71	3.54	
1.03	6.02	5.68	
2.05	9.47	8.92	
4.94	19.24	19.38	
9.80	35.12	37.56	
19.42	58.07	61.95	
51.62	87.56		
102.3	95.79	95.28	
202.8	99.03	98.49	
200 R	99 94	99.75	

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PTL also reported a moisture content of 67.3% for the centrifuge sediment sample. Based upon this moisture content and the wet weight of the sample, a total of approximately 21 grams of dry solids were captured during the 50 minute demonstration.

Based on the mass of dry solids collected (approximately 21 grams) and volume of water centrifuged (assume 20 gallons), the TSS level in the 400-gallon tank was approximately 300 mg/L. Note that this TSS level was greater than those previously measured for river samples (50-200 mg/L) and CSO samples (approximately 100 mg/L).

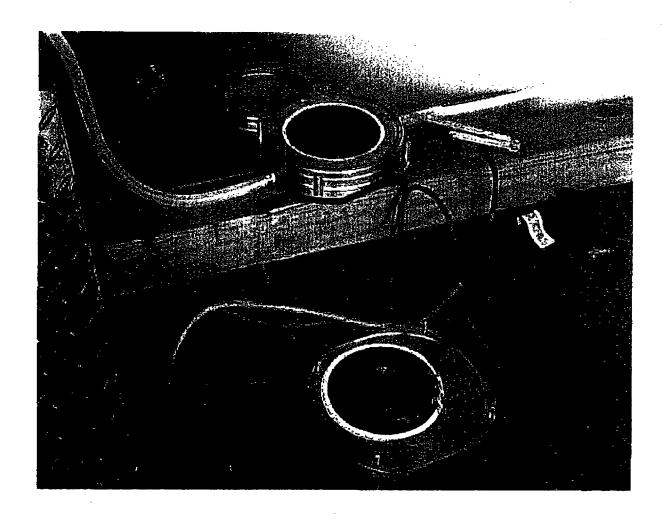
Digital photos taken of the filtered solids and centrifuge sample collection are attached.

REH/reh

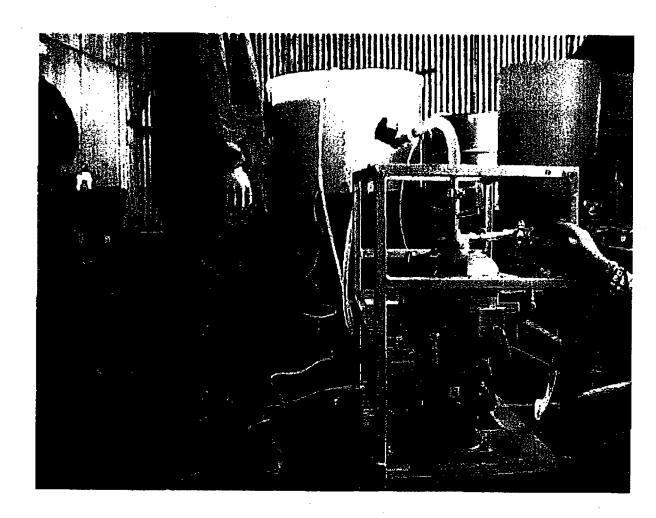
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MVC-005S Filter Collection - Multiplate filtration unit with 1.2um filters



MVC-015S Centrifuge Demonstration - Centrifuge system - Passaic River water demo



MVC-029S Centrifuge Demonstration - 250 ml jar with 64 grams solids (wet wt.) after 50 min. of centrifuging

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TABLE
COMPARISON OF RESULTS
Centrifugation versus Filtration

	CENTRIFUGATION	FILTRATION
Grain Size Distribution		, in regional as maken
Particle Size (um)	Volume under (%)	Volume under (%)
0.522	3.71	3.54
1.03	6.02	5.68
2.05	9.47	8.92
4.94	19.24	19.38
9.80	35.12	37.56
19.42	58.07	61.95
51.62	87.56	88.30
102.3	95.79	95.28
202.8	99.03	98.49
299.8	99.94	99.75
Other Variables	-	
Moisture Content	~60%	~90%*
Time to process 400 gallons	~16 hours	~40 hours

• The 90% moisture content result is based on the Trial Run filtration results, not the comparison test results; only the centrifuged sample was analyzed for moisture content.

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1c: Section Chief
NJDEP-Bureau of Federal Case Management
401 East State Street -CN 028
Trenton, NJ 08625-0028
Attn: Jonathan D. Berg

1c: Chief, New Jersey Superfund Branch
Office of Regional Counsel
U.S. Environmental Protection Agency
290 Broadway, 19th Floor, Room W-20
New York, NY 10007-1866
Attention: Diamond Alkali Site Attorney - Passaic River Study Area

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ECOLOGICAL SAMPLING PLAN FOR THE PASSAIC RIVER STUDY AREA

STANDARD OPERATING PROCEDURE NO. 6

COMBINED SEWER OVERFLOW SAMPLING

September 2000

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1.0 APPLICABILITY

This Standard Operating Procedure (SOP) defines one of the procedures which will be followed for the collection and preparation of combined sewer overflow (CSO) effluent and solids samples from the Passaic River Study Area (Study Area). These procedures give descriptions of equipment and field procedures necessary to collect CSO samples in this manner. SOP-14 provides an alternate method for collection of CSO solids.

Other SOPs that will be utilized with this procedure include:

- SOP No. 1 Decontamination
 - SOP No. 2 Containers, Preservation, Handling and Tracking of Sample
 - SOP No. 5 Surface Water Quality Physicochemical Parameters

2.0 PREPARATIONS FOR SAMPLING

The FSP identifies the process for the selection of CSO sampling stations, the frequency of sampling, sample type, field measurements, and analytical procedures for this study element. The field team is responsible for reviewing the FSP prior to conducting field activities and ensuring that all field equipment, including sample containers and preservatives, are available and in acceptable condition.

3.0 FIELD EQUIPMENT LIST

Equipment to be used during the collection of CSO effluent samples (including water and solids) will depend on the collection methodologies selected in this SOP. Equipment may include, but is not limited to, the following:

- 900 Lite Sigma Stormwater Sampler with built-in rain gauge, integral flow meter, and electronic depth sensor, temperature/pH meter, dissolved oxygen/conductivity meter (or equivalent), and a maximum pump flow rate of 1500 ml/min at 3 ft vertical lift
- Backup temperature/pH meter, and dissolved oxygen/conductivity meter
- Field monitoring equipment (OVA or HNu, and CGI/O₂ meter)

- Sample collection containers
- Preservatives (as required)
- 6 gallon (23 liter) polyethylene container
- 300-400 gallon trailer-mounted polyethylene holding tank
- Westfalia Centrifuge (modified by Machine All, Inc., Ontario, Canada)
- Submersible pump (stainless steel/Teflon)
- Filtration unit
- 1.2 um glass fiber filters
- Peristaltic or centripetal pump
- Teflon (or equivalent) tubing
- Spill containment container/pan
- Laboratory sample containers
- Wet Icc
- Insulated coolers
- Sample identification labels
- Tape
- Waterproof marking pens
- Sealable plastic bags and bubble wrap
- Litmus test strips for confirming proper sample preservation
- Power mixer (for 400 gallon tank; stainless steel blade)
- Decontamination equipment

4.0 DECONTAMINATION OF EQUIPMENT

Decontamination of CSO sampling equipment will be performed prior to the initial set-up and between each sampling event at each location in accordance with procedures outlined in SOP No. 1 - Decontamination. Personnel decontamination procedures are contained in the HASP.

5.0 LOCATION OF SAMPLING STATIONS

The selection process of the CSOs to be sampled is described in the FSP. Once on location, the manhole access where the samples will be collected will be established and labeled on a map.

6.0 CSO SAMPLE COLLECTION AND PREPARATION

The following procedure shall be implemented, as practicable, for collecting CSO samples from operable and accessible CSOs that discharge into the Study Area. The analytical sample volume requirements for the aqueous effluent samples and the solids samples are provided in Tables 4-1 and 4-2 of the QAPP, respectively. Samples will be preserved in the field following compositing (described below). The following list presents the procedures to be followed during CSO effluent and CSO solid sample collection, preservation and processing.

6.1 Automated Sampling Procedure

- 1. The automated sampling equipment (if used) will be securely mounted either 1) inside the manhole that is selected for sampling, 2) inside the CSO discharge pipe, or 3) in a protected location adjacent to the discharge pipe with the equipment probes secured inside the CSO. A 6 gallon polyethylene container will be set up inside the housing of the portable sampler. This container will be used to collect a 30 minute continuous composite of the first-flush, and allow sufficient time for mobilization of the 300-400 gallon holding tank to the CSO location for additional sample collection.
- 2. Check automated sampler calibrations/settings and monitoring equipment calibrations/settings per operation manuals (as appropriate).
- 3. Record the sampling location position and the depth of the water within the CSO pipe at the sampling location (if appropriate) in the field logbook.
- 4. Program the automated sampler to begin sampling when an internal flow/depth sensing device (which includes an area-velocity flow meter coupled to the sampling system and a probe that is in the channel beside the intake tubing) indicates an increase in positive flow and/or depth. Program the system to recognize and not sample negative flow (i.e, backflow) which may result from tidal influence and river water entering the outfall.

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- 5. Initiate the autosampler modem/personal beeper equipment to notify a designated field team member when the sampler begins collection during an overflow event.
- 6. Following notification by the autosampler modem that collection has started, mobilize the trailer mounted with the 300 400 gallon capacity polyethylene container (hopper bin) to the CSO location. This holding tank will be opened on top and have a ball valve on the bottom which can be used for sample collection.
- 7. Transfer the discharge tubing from the 6 gallon polyethylene container in the manhole to the larger holding tank on the trailer. Empty the 6 gallon sample container into the larger holding tank. Continue pumping until the end of the overflow event or until the holding tank is full. If the sample container is filled prior to the end of the overflow event, flow, pH, temperature, dissolved oxygen, and conductivity will continue to be automatically monitored for the duration of the storm.
- 8. Transport the container to the staging area. Collect aqueous effluent samples for chemical analyses following procedures outlined in Section 6.3.

6.2 Alternative Manual Sampling Procedure

This alternative manual procedure may be required if an automated sampler is not used, or if there is pump or other system failure of the automated sampler.

Measure and record temperature, dissolved oxygen, salinity/conductivity, and pH in the discharging water from the CSO pipe in accordance with SOP No. 5 - Surface Water Physicochemical Parameters. These measurements can be made directly from the pipe or a grab sample. The frequency of these measurements should be a minimum of every fifteen minutes unless field conditions limit the field teams' ability to sample at that frequency.

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- 2. Initiate flow and depth measurement at the first indication of overflow. This information will be collected every 15 minutes during sample collection and logged in the field logbook.
- 3. Set the submersible pump (or equivalent) in the water column of the CSO pipe to initiate sample collection with the first flush and continue to sample until the 300-400 gallon holding tank is full, or the storm event is over.
- 4. Transport the holding tank to the staging area. Collect aqueous effluent samples for chemical analyses following procedures outlined in Section 6.3.

6.3 CSO Effluent Sample Preparation

Upon arrival at the staging area or an appropriately identified staging area the CSO effluent samples will be prepared for shipment to the analytical laboratory(s). Both unfiltered and field filtered samples will be collected for analysis. The procedure for this sample collection is outlined below.

- 1. Homogenize the effluent sample in the holding tank.
- 2. Collect effluent samples from the 300-400 gallon holding tank using a decontaminated beaker, bailer, or other sampling equipment.
- 3. Transfer to the appropriate laboratory sample containers by gently pouring the collected sample into the appropriate containers by allowing the sample to flow slowly down the interior of the laboratory sample containers. The sample bottles should be alternated during the filling process.
- 4. Add preservative to laboratory sample containers as specified in Table 4-1 of QAPP.
- Label the sample containers with the appropriate pre-printed sample identification labels.
- 6. Close and scal each container. Proceed with Step No. 8 for the unfiltered samples.

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- 7. Transfer samples for filtering into the hand pump at an appropriate rate to avoid overflow. Pump the appropriate sample volume through a 0.45 micron glass fiber filter(s). Transfer the filtrate into the appropriate laboratory sample container and label with the appropriate pre-printed sample identification labels. Discard the filters.
- 8. Check label on each sample container and cover the label with clear plastic tape.
- 9. Wrap sample containers in bubble wrap and place in a scalable plastic bag. Place bag in refrigerator until sample is ready for packing and shipping.
- 10. Complete the appropriate chain-of-custody form for each sample container, and seal in plastic bag.
- 11. Pack samples in shipping cooler with ice. Tape completed chain-of-custody form on the inside of the lid of the shipping cooler and seal cooler with tape and custody seals. Ship within 24 hours of collection by overnight courier to the appropriate laboratory.

6.4 **CSO Settleable Solids Collection**

- 1. Allow the suspended solids to settle in the holding tank undisturbed for up to 48 hours.
- 2. Collect settled solid samples from bottom of tank using a shovel or equivalent sediment collection equipment. If insufficient solids are collected, filter the effluent following the procedures described in Section 6.5.
- 3. Transfer solids to a decontaminated tared glass or stainless steel container and weigh on an electronic balance capable of weighing to the nearest 0.1g. Remove any nonsediment debris, weigh and determine whether sufficient mass has been collected, as required in Table 2-5 of the FSP. Continue removing settled solids from composite tank until the target mass has been reached. Homogenize the solids.

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- 4. If sufficient mass has been collected, transfer the settled solids to the appropriately labeled containers. Close and seal each container.
- 5. Check the label on each sample container and cover the label with clear plastic tape.
- 6. Wrap sample containers in bubble wrap and place in a sealable plastic bag. Place bag in refrigerator until sample is ready for packing and shipping.
- 7. Complete the appropriate chain-of-custody form for each sample container, and seal in plastic bag.
- 8. Pack samples in shipping cooler with wet ice. Tape completed chain-of-custody form on the inside of the lid of the shipping cooler and seal cooler with tape and custody seals. Ship within 24 hours of collection by overnight courier to the appropriate laboratory.

6.5 CSO Solids Collection from CSO Effluent

If an adequate quantity of solids are not collected via settling (as described in Section 6.4), additional solids will be obtained from the CSO effluent using either centrifugation or filtration methodologies. The centrifuge methodology will be the preferred methodology, if feasible. Solids collection protocols for both methodologies are detailed below.

6.5.1 Centrifugation Methodology

- 1. Mix CSO effluent in 400 gallon tank with power mixer; continue mixing throughout centrifugation process.
- 2. Transfer the appropriate volume of homogenized CSO effluent to the TSS and TOC sample containers. Add preservatives to the TOC container, label, and seal. Place in refrigerator or cooler until ready for shipment.

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- 3. Label the appropriate sample containers with the pre-printed sample identification labels.
- 4. Set centrifuge on level surface to ensure stability. Disassemble bowl components.
- 5. Decontaminate the centrifuge bowl components in accordance with the procedures outlined in SOP No. 1 Decontamination.
- 6. Reassemble the centrifuge bowl components and reattach to the centrifuge in accordance with the Machine All operation manual. Make sure that the drain spigot is not near the electrical panel. Tape the spring clamps in place to prevent loosening due to vibration. Attach the drain lines and ensure that the centrifuge has been checked for any loose materials, etc.
- 7. Place the submersible pump in the 400-gallon tank. Do not hook the pump to the centrifuge yet.
- 8. Place the outlet hose from the pump over the rim of the 400-gallon tank. Start the pump and adjust the valve on the water line to achieve a flow rate of 1-2 liters per minute. Turn the pump off and secure the outlet hose to the centrifuge.
- 9. Start the centrifuge and wait until the machine is up to speed. When the centrifuge is at speed, turn the pump on and watch for the water to exit the drain pipe. The back-pressure valve should be adjusted so that there are no bubbles in the sight glass on the discharge pipe.
- 10. Record the flow rate every 10 15 minutes and adjust as required.
- 11. At the completion of centrifugation, turn off the pump and disconnect the outlet hose from the centrifuge. After all water has stopped flowing from the centrifuge discharge pipe, turn the centrifuge off.

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- 12. Remove the bowl and take to a flat, solid, clean area to disassemble. Remove the centrifuge bowl pump and, without taking the bowl apart, drain off all remaining water from the bowl into a decontaminated squeeze bottle, with excess water stored in a decontaminated sample bottle. This water will be used to (1) rinse material from the bowl assembly and (2) provide additional material, if needed, by drying.
- 13. Disassemble the bowl, placing each section inside the other so as not to cause any contamination.
- 14. Clean each section starting with the innermost triangular section and work outward. Use a Teflon spatula to remove solids from the bowl surfaces into a decontaminated stainless steel or glass container. After loosening the material with a brush and spatula, use the squeeze bottle to sparingly wash each section into a separate decontaminated container. After all sections have been cleaned, pour all of the water captured from the bowl and squeeze bottle into a sample container.
- 15. Properly decontaminate the bowl pieces in accordance with SOP 1 -Decontamination and store all equipment appropriately.
- 16. Weigh the collected solids/sample container to determine whether sufficient mass has been collected, as required in Table 2-5 of the FSP. Continue centrifugation until the target mass has been reached.
- **17**. When sufficient mass has been collected, homogenize and transfer the collected solids to the appropriately labeled containers. Close and seal each container. If, at the conclusion of centrifugation of the whole 400 gallons, insufficient mass has been collected, prepare additional sample containers with the retained rinse water to be submitted to the laboratories for drying.

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- 18. Check the label on each sample container and cover the label with clear plastic tape.
- 19. Wrap sample containers in bubble wrap and place in a scalable plastic bag.

 Place bag in refrigerator until sample is ready for packing and shipping.
- 20. Complete the appropriate chain-of-custody form for each sample container, and seal in plastic bag.
- 21. Pack samples in shipping cooler with ice. Tape completed chain-of-custody form on the inside of the lid of the shipping cooler and seal cooler with tape and custody seals. Ship within 24 hours of collection by overnight courier to the appropriate laboratory.

6.5.2 Filtration Methodology

- 1. Mix CSO effluent in the 400-gallon tank with power mixer; continue mixing throughout filtering process.
- 2. Transfer the appropriate volume of homogenized CSO effluent to the TSS and TOC sample containers. Add preservatives to the TOC container, label, and seal. Place in refrigerator or cooler until ready for shipment.
- 3. Label the appropriate sample containers with the pre-printed sample identification labels.
- 4. Place the decontaminated 1.2 μm filter in the filtering apparatus. Start the vacuum or hand pump and add an appropriate volume of homogenized effluent sample.
- Continue to add effluent sample until the filtrate container is full, or the
 filtering time slows, whichever comes first. Carefully remove the filter with
 forceps and place on a decontaminated glass plate. Retain 50-mL of filtrate

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for apparatus rinse, and decant the remaining filtrate into a container for subsequent disposal. Add a new filter and resume filtration.

- 6. If the filtering process is slow, or visual inspection of the effluent suggests that there is a large concentration of suspended solids, sequential filtration may be required. In this case, the effluent is initially passed through the 10 μm filters, followed by filtering with the 1.2 μm filter.
- 7. Rinse the filtering apparatus with the retained filtrate to ensure that all of the solids have been removed from the sides of the filter holder.
- 8. Scrape the solids from the filters, using a decontaminated stainless steel spatula, onto a tared glass plate, taking care to not remove any filter media. Remove any non-sediment debris, weigh and determine whether sufficient mass is collected. Continue filtering until the target mass has been reached.
- 9. If sufficient mass has been collected, homogenize, and transfer the filtered sediment to the appropriately labeled containers. Close and seal each container.
- Check the label on each sample container and cover the label with clear plastic tape.
- 11. Wrap sample containers in bubble wrap and place in a sealable plastic bag. Place bag in refrigerator until sample is ready for packing and shipping.
- 12. Complete the appropriate chain-of-custody form for each sample container, and seal in plastic bag.
- 13. Pack samples in shipping cooler with ice. Tape completed chain-of-custody form on the inside of the lid of the shipping cooler and seal cooler with tape and custody seals. Ship within 24 hours of collection by overnight courier to the appropriate laboratory.

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7.0 SAMPLE CONTAINERS, PRESERVATION, HANDLING AND TRACKING

Sample containers, handling and preservation procedures are described in SOP No. 2 - Containers, Preservation, Handling and Tracking of Samples for Analysis.

8.0 QUALITY CONTROL SAMPLES

To identify potential sources of sample contamination and evaluate potential error introduced by sample collection and handling, field quality control samples (QC samples) will be collected during the CSO sample collection. All QC samples will be labeled in accordance with SOP No. 2 - Containers, Preservation, Handling and Tracking of Samples, and sent to the laboratory with the other samples for analysis. QC samples for CSO sampling will include rinsate samples, field duplicate samples, and matrix spike samples and will be collected at the frequency identified in the QAPP.



 $To: \ensuremath{\mathsf{Rick}} \ensuremath{\mathsf{WInfield}}$

Voice Phone Number:

From: Clifford E. Firstenberg

Company: Firstenberg Consulting LLC

Fax Number: 757 258 7721

Voice Number: 757 258 7720

MERCACE

MESSAGE				
30 pages including cover				
+++++++++++++++++		•		
Rick				•
Here's the letter regarding the	he centrifuge test and C	LH's request that EPA	approve the new method.	
There are a couple of color discuss what these show.	picture which will prbabl	y not be very useful aft	ter being faxedplease cat	if you want to
Cliff				
			•	

Date: 10/16/01 Pages: 1 of 1

Particle Technology Labs, Ltd.



630/969-2703 • FAX 630/969-2745 FDA Certificate# 1422692



BLASLAND BOUCK & LEE, INC. 6723 Towpath Road
Syracuse, NY 13214

Attn: Mr. Russell Houck

Subject: Particle Size Analysis and Moisture Content of Two Particulate Samples

PTL Project: 4395

Dear Mr. Houck:

Enclosed are the results from the particle size analysis and moisture content conducted on your two Particulate samples received 5/22/00. The particle size was determined on our Malvern Mastersizer S LASER diffractor. For your Filters with sediment, one filter was selected at random and wetted with Triton X-100 and sodium pyrophosphate in water. The particulate was collected in a large beaker and ultrasonicated for a timed two minutes. An aliquot portion of the Centrifuged solids sample was wetted with Triton X-100 and sodium pyrophosphate in water and ultrasonicated for a timed two minutes. Both samples were analyzed according to standard operating procedures for this instrumentation type.

Please note that these samples were received in a very clumpy, agglomerated state. Therefore, the addition of wetting agents and the application of ultrasonic energy were used to aid in sample dispersion. Following the dispersion process, microscope examination revealed that these samples still contained floccy agglomerates which did not break apart during dispersion. These results reflect the inclusion of the floccy agglomerates.

An explanation page entitled, "INTERPRETING YOUR MALVERN FINE PARTICLE ANALYSIS," has also been included for your review.

At you request, the moisture content was also determined for the Centrifuged Solids sample. The moisture content was determined to be:

Moisture Content: 67.3%

We trust this information will be beneficial for your future use. If there are any questions concerning this data or the methods used to acquire the data, please do not hesitate to contact us here at Particle Technology Labs.

Submitted by,

Amy Hihn

Reviewed by, Teller

DR:\4395.doc:391

Particle Technology Labs, Ltd.

555 ROGERS STREET • DOWNERS GROVE, ILLINOIS 60515
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INTERPRETING YOUR MALVERN FINE PARTICLE ANALYSIS

INTRODUCTION

Your samples have been analyzed on a Malvern MasterSizer S LASER particle size analyzer. This instrument analyzes particles on an ensemble basis, calculating a volume distribution from the LASER diffraction pattern of particle diameters.

THE REPORT

The information presented in your report consists of four basic categories:

SAMPLE DETAILS: The first section of the report page consists of SAMPLE DETAILS: client, sample identification, and sample conditioning information. If you find it necessary to contact us concerning this work, please refer to the four-digit PTL Project #, which appears under Sample File.

SYSTEM DETAILS: This section describes parameters specific to the instrument being used as well as to the analysis. It should be noted that in the size region of 0.1 - 3 microns, LASER technology is sensitive to the refractive index (RI) of the sample material. Therefore, a standard instrument default value for the refractive index of the sample and carrier fluid is normally used if one is not provided by the client. A sample RI value of 1.5295 (glass beads) is used along with a carrier fluid RI of 1.33 (water). Likewise, the Presentation: 0HD and Analysis Model: Polydisperse is used as a default. For samples analyzed on a dry basis, the particle RI is 1.45 with a dispersant RI of 1.00 (air). Correspondingly, the Presentation: RHA is used as the default. If the client provides a RI at a later date, the raw data can be recalculated to adjust for the refractive index.

RESULT STATISTICS: This section presents

- 1. Statistics
 - A. ***Statistics ALWAYS appear as "Percent Less Than" or "% Under."***
 - B. Span is defined as the 90% 10% size divided by the 50% size level.
 - C. The Surface to Volume mean diameter is reported as the D[3,2] value.
 - D. The Mean Volume statistic is reported as the D[4,3] value.
 - E. The 90%, 50%, and the 10% size values appear as D[v,0.90], D[v,0.50] and D[v,0.10] respectively.
 - F. A calculated Specific Surface Area value is reported. However, consider this value only as an approximate surface area since calculations are based upon smooth spheres.
- 2. Tabulated data as a Cumulative Weight Percent Greater Than (Or Less Than) Indicated Size.

HISTOGRAM: This last section displays the differential histogram as well as the cumulative curve for the data displayed in the table above.

DR:\Malvem S Dry Interp Sheet:215

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MASTERSIZER

Result: Histogram Report

Sample ID: FILTERSED01

Sample File: 4395

Sample Path: A:\

Sample Notes: BLASLAND 80UCK & LEE

Carrier: Water Dispersant Na Pyrophos., Triton X-100

Ultrasonic 2 minutes Particle Technology Labs

Sample Details Run Number: 2 Record Number: 2

Filters with Sediment

Tech: AH PTL: 24297

System Details

Beam Length: 2.40 mm

[Particle R.I. = (1.5295, 0.1000);

Dispersant R.I. = 1.3300]

Sampler: MS1

Obscuration: 27.7 %

Residual: 0.442 %

Analysis Model: Polydisperse Modifications: None

Range Lens: 300RF mm

Presentation: 3OHD

Distribution Type: Volume

Mean Diameters: D [4, 3] = 26.83 um

Result Statistics Concentration = 0,0267 %Voi Density = 1,000 g / cub. cm

D (v, 0.1) = 2.21 um

Specific S.A. = 1.6175 sq. m/g

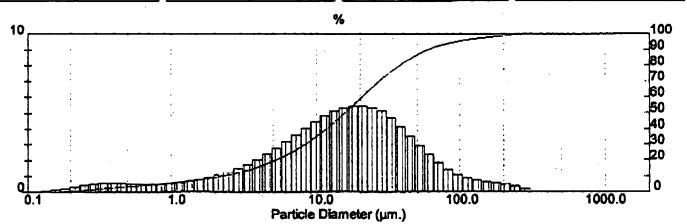
Measured: Tue May 23 2000 9:52AM

Analysed: Tue May 23 2000 9:52AM

Result Source: Analysed

D (v, 0.9) = 59.24 um D (v, 0.5) = 15.48 um Uniformity = 1.287E+00 $D[3, 2] = 3.71 \, \text{um}$ Span = 3.685E+00

1	5 ize	Volume	Size	Volume	Size	Volume	528	Volume
1.	(um)	Under %	(um)	Under %	(um)	Under %	(um)	Under %
Γ	0.055	0.00	0.635	4.38	7.31	27,26	84.15	94.29
1	0.061	0.00	0.700	4.69	8.06	29.71	92.79	95.10
1	0.067	0.00	0.772	5.01	8.89	32.33	102.3	95.79
1	0.074	0.01	0.851	5.33	9.80	35.12	112.8	96.39
1	0.082	0.01	0.938	5.67	10.81	38.06	124.4	96.93
1	0.090	0.02	1.03	6.02	11.91	41.15	137.2	97.41
1	0.099	0.03	1.14	8.40	13.14	44.37	151.3	97.88
1	0.109	0.05	1.26	6.81	14.49	47.70	166.8	98.28
1.	0.121	0.07	1.39	7.25	15.97	51,11	183.9	98.67
1	0.133	0.11	1.53	7.74	17.62	54.58	202.8	99.03
1.	0.147	0.17	1,69	8.26	19.42	58.07	223.6	99.35
1	0.162	0.25	1.86	8.84	21.42	B1.56	246.6	99.61
1	0.178	0.36	2.05	9.47	23.62	65.01	271.9	99.52
1	0.196	0.50	2.26	10.17	26.04	68.41	299.8	99.94
	0.217	0.69	2.49	10.94	28.72	71.78	330.6	99.98
1.	0.239	0.93	2.75	11.79	31.66	74.96	364,6	100.00
ľ	0.263	1.22	3.03	12.73	34.92	77.95	402.0	100.00
	0.290	1.55	3.34	13.78	38.50	80.72	443.3	100.00
	0.320	1.91	3.69	14.94	42.45	83.25	488.8	100.00
ŀ	0.353	2,28	4.07	16.23	46.81	85.53	539.0	100.00
ŀ	0.389	2.66	4.48	17.66	51.62	87.58	594.3	100.00
1	0.429	3.01	4.94	19.24	56.92	89.34	655.4	100.00
1	0.473	3.37	5.45	20.99	62.76	90.88	722.7	100.00
1.	0.522	3.71	6.01	22.91	69.21	92.21	796.9	100.00
ľ	0.576	4.05	6.63	25.00	76.32	93.34	878.7	100.00



Malvem Instruments Ltd. Malvern, UK

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Mastersizer S long bed Ver. 2.18 Serial Number:

23 May 00 14:55

Page 29/30 :MA32:8 10-81-300

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geuf By: Firstenberg Consulting LLG;



Result: Histogram Report

Sample Details

Sample ID: CENTSED01 Sample File: 4395

Run Number: 2 Record Number: 6 Measured: Tue May 23 2000 10:33AM Analysed: Tue May 23 2000 10:33AM

Result Source: Analysed

Sample Path: A:\

Sample Notes: BLASLAND BOUCK & LEE

Carrier: Water Dispersant: Na Pyrophos., Triton X-100

Centrifuged Solids

Ultrasonic: 2 minutes

Tech: AH

Particle Technology Labs

PTL: 24296

Range Lens: 300RF mm

Beam Langth: 2.40 mm

Sampler, MS1

Obscuration: 28.5 %

Presentation: 30HD

[Particle R.I. = (1.5295, 0.1000);

Dispersant R.I. = 1.3300)

Residual: 0.385 %

Analysis Model: Polydisperse

Distribution Type: Volume

Modifications: None

Result Statistics

System Details

Concentration = 0.0275 %Vol

Density = 1.000 g / cub. cm $D(v, 0.5) = 13.96 \mu m$

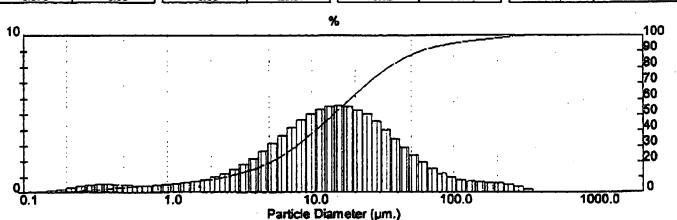
Specific S.A. = 1.5892 sq. m/g

Mean Diameters: D [4, 3] = 28.75 um D (v, 0.1) = 2.38 um D [3, 2] = 3.78 um

Span = 3.971E+00

 $D(v, 0.9) = 57.84 \, \text{um}$ Uniformity = 1.452E+00

Size	Volume	Size	Volume	Size	Volume	Size	Valume
(um)	Under %						
0.055	0.00	0.638	4.16	7.31	28.58	84.15	93.95
0.061	0.00	0.700	4.45	8.08	31.39	92.79	94.66
0.067	0.00	0.772	4.74	8.89	34.39	102.3	95.28
0.074	0.01	0.851	5.04	9.80	37.56	112.8	95.83
0.082	0.01	0.938	5.35	10.81	40.87	124.4	96.33
0.090	0.02	1.03	5.68	11.91	44,29	137.2	96.79
0.099	0.03	1,14	6.02	13.14	47.79	151.3	97.24
0.109	0.04	1.26	6.40	14,49	51.34	166.8	97.67
0.121	0.07	1.39	6.81	16,97	54.91	183.9	98.09
0.133	0.11	1.53	7.26	17.62	58,46	202.8	98.49
0.147	0.18	1.69	7.76	19.42	61.95	223.6	98.88
0.182	0.24	1.86	8.31	21.42	65.37	246.6	99 <i>.2</i> 3
0.178	0.34	2.05	8.92	23.62	68.70	271.9	99.52
0.196	0.48	2.26	9.60	25.04	71,91	299.8	99.75
0.217	0.86	2.49	10.38	28.72	74.92	330.6	99.95
0.239	0.89	2.75	11.25	31.66	77.70	364.6	100.00
0.263	1.17	3.03	12.23	34,92	80.27	402.0	100.00
0,290	1,49	3.34	13.34	38.50	82.61	443.3	100,00
0.320	1.83	3.69	14.59	42,45	84.73	488.8	100.00
0.353	2.18	4.07	16.01	46,61	86.62	539.0	100.00
0.389	2,53	4.48	17.60	51.62	88.30	594.3	100.00
0.429	2.88	4.94	19.38	56.92	89.78	655.4	100.00
0.473	3.21	5.45	21.37	62.76	91.06	722.7	100.00
0.522	3.54	6.01	23.57	69.21	92.17	796.9	100.00
0.576	3.66	6.63	25.97	76.32	93.13	878.7	100.00



laivem instruments Ltd. laivern, UK

Mastersizer S long bed Ver. 2.16 Serial Number:

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